#### CETIFICATION

SDG No:

MC46948

Laboratory:

Accutest, Massachusetts

Site:

BMSMC, Phase 2A Release

Matrix:

Groundwater

Assessment, Humacao, PR

Humacao, PR

**SUMMARY:** 

Groundwater samples (Table 1) were collected on the BMSMC facility – Phase 2A Release Assessment Area. The BMSMC facility is located in Humacao, PR. Samples were taken July 17-20, 2016 and were analyzed in Accutest Laboratory of Marlborough, Massachusetts that reported the data under SDG No.: MC46948. Results were validated using the following quality control criteria of the methods employed (MAPED EPH, Massachusets Department of Environmental Protection, 2004) and the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE	MATRIX	ANALYSIS PERFORMED
	DESCRIPTION		
MC46948-1	OSGP11-GWD	Groundwater	Extractable TPHC Ranges
MC46948-2	OSGP11D-GWD	Groundwater	Extractable TPHC Ranges
MC46948-3	OSGP11-GWS	Groundwater	Extractable TPHC Ranges

CO I CEN

Reviewer Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

August 2, 2016

A 1591674

### **SGS** Accutest

# Report of Analysis

Page 1 of 1

Client Sample	ID: OSGP11-GWD
Lab Sample II	): MC46948-3
Matrix:	AO - Ground W.

/ater MADEP EPH REV 1.1 SW846 3510C Date Sampled: 07/20/16 Date Received: 07/21/16 Percent Solids: n/a

Method: Project:

BMSMC Phase 2A Release Assessment, Humacao, PR

File ID DF Analyzed Ву Prep Date Prep Batch **Analytical Batch** Run #1 DE14946.D 1 07/25/16 TA 07/21/16 OP48223 **GDE832** 

Run #	2
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	Initial Volume	Final Volume
Run #1	870 ml	2.0 ml

Run	#2

CAS No.	Compound	Result	RL	MDL	Units	Q
83-32-9	Acenaphthene	ND	5.7	1.8	ug/l	
208-96-8	Acenaphthylene	ND	5.7	0.41	ug/l	
120-12-7	Anthracene	ND	5.7	0.67	ug/l	
56-55-3	Benzo(a)anthracene	ND	5.7	0.35	ug/l	
50-32-8	Benzo(a)pyrene	ND	5.7	0.34	ug/l	
205-99-2	Benzo(b)fluoranthene	ND	5.7	0.51	ug/l	
191-24-2	Benzo(g,h,i)perylene	ND	5.7	0.43	ug/l	
207-08-9	Benzo(k)fluoranthene	ND	5.7	0.41	ug/I	
218-01-9	Chrysene	ND	5.7	0.50	ug/l	
53-70-3	Dibenz(a,h)anthracene	ND	5.7	0.45	ug/l	
206-44-0	Fluoranthene	ND	5.7	0.38	ug/f	
86-73-7	Fluorene	ND	5.7	0.46	ug/l	
193-39-5	Indeno(1,2,3-cd)pyrene	ND	5.7	0.34	ug/l	
91-57-6	2-Methylnaphthalene	ND	5.7	0.52	ug/l	
91-20-3	Naphthalene	ND	5.7	1.1	ug/l	
85-01-8	Phenanthrene	ND	5.7	0.35	ug/l	
129-00-0	Pyrene	ND	5.7	0.69	ug/l	
	C11-C22 Aromatics (Unadj.)	33.6	110	33	ug/l	JB
	C9-C18 Aliphatics	25.2	110	19	ug/l	JB
	C19-C36 Aliphatics	58.5	110	31	ug/l	j
	C11-C22 Aromatics	33.6	110	33	ug/l	JВ

CAS No.	Surrogate Recoveries	Run#1	Run# 2	Limits
84-15-1	o-Terphenyl	54%		40-140%
321-60-8	2-Fluorobiphenyl	64%		40-140%
3386-33-2	1-Chlorooctadecane	50%		40-140%
580-13-2	2-Bromonaphthalene	69%		40-140%



ND = Not detected

MDL = Method Detection Limit

J = Indicates an estimated value

RL = Reporting Limit

B = Indicates analyte found in associated method blank

E = Indicates value exceeds calibration range

N = Indicates presumptive evidence of a compound

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MC46948: Chain of Custody Page 1 of 2

#### SGS Accutest

# Report of Analysis

Page 1 of 1

Client Sample ID:	OSGP11-GWS
Lab Sample ID:	MC46948-1
Mateir	AO Cround W

Initial Volume

890 ml

AQ - Ground Water

Date Sampled: 07/19/16 Date Received: 07/21/16

Method:

MADEP EPH REV 1.1 SW846 3510C

Final Volume 2.0 ml

Percent Solids: n/a

Project:

Run #1

BMSMC Phase 2A Release Assessment, Humacao, PR

Run #1	File ID DE14944.D	DF 1	Analyzed 07/25/16	Ву	Prep Date 07/21/16	Prep Batch OP48223	Analytical Batch GDE832
Run #2 a	DE14962.D	1	07/26/16	TA	07/21/16	OP48223	GDE833

Run #2	890 ml 2.0 ml					
CAS No.	Compound	Result	RL	MDL	Units	(
83-32-9	Acenaphthene	ND	5.6	1.8	ug/l	
208-96-8	Acenaphthylene	ND	5.6	0.40	ug/l	
120-12-7	Anthracene	ND	5.6	0.65	ug/l	
56-55-3	Benzo(a)anthracene	0.45	5.6	0.34	ug/l	J
50-32-8	Benzo(a) pyrene	ND	5.6	0.33	ug/I	
205-99-2	Benzo(b)fluoranthene	ND	5.6	0.50	ug/l	
91-24-2	Benzo(g,h,i)perylene	ND	5.6	0.42	ug/I	
207-08-9	Benzo(k)fluoranthene	ND	5.6	0.40	ug/l	
218-01-9	Chrysene	ND	5.6	0.49	ug/l	
3-70-3	Dibenz(a,h)anthracene	ND	5.6	0.44	ug/l	
06-44-0	Fluoranthene	ND	5.6	0.38	ug/l	
6-73-7	Fluorene	ND	5.6	0.45	ug/l	
93-39-5	Indeno(1,2,3-cd)pyrene	ND	5.6	0.33	ug/l	
1-57-6	2-Methylnaphthalene	ND	5.6	0.51	ug/l	
1-20-3	Naphthalene	ND	5.6	1.1	ug/l	
5-01-8	Phenanthrene	ND	5.6	0.34	ug/l	
29-00-0	Pyrene	ND	5.6	0.67	ug/l	
	C11-C22 Aromatics (Unadj.)	40.5	110	32	ug/l	J
	C9-C18 Aliphatics	19.7	110	19	ug/l	J
	C19-C36 Aliphatics	36.5	110	30	ug/l	j
	C11-C22 Aromatics	38.2	110	32	ug/l	J
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
84-15-1	o-Terphenyl	65%	57%	40-1	40%	
21-60-8	2-Fluorobiphenyl	79%	62%	40-1	40%	
386-33-2	1-Chlorooctadecane	35% b	36% b	40-1	40%	1
80-13-2	2-Bromonaphthalene	85%	70%		40%	

ND = Not detected

MDL = Method Detection Limit

J = Indicates an estimated value

RL = Reporting Limit

B = Indicates analyte found in associated method blank

Parfael Infl Méndez LIC # (88

E = Indicates value exceeds calibration range

N = Indicates presumptive evidence of a compound

<sup>(</sup>a) Confirmation run.

<sup>(</sup>b) Outside control limits due to possible matrix interference. Confirmed by refractionation/reanalysis.

### **SGS** Accutest

# Report of Analysis

Page 1 of 1

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Client Sam Lab Samp Matrix: Method: Project;	le ID: MC469 AQ - G MADE	1D-GWS 48-2 round Water P EPH REV 1 C Phase 2A R			nacao, P	Date Perc	-	7/19/16 7/21/16 /a
Run #1 Run #2	File ID DE14945.D		<b>Analyzed</b> 07/25/16	By TA	Prep D 07/21/		Prep Batch OP48223	Analytical Batch GDE832
Run #1 Run #2	Initial Volume 880 ml	Final Volu 2.0 ml	ne					
CAS No.	Compound		Result	RL	MDL	Units	Q	
83-32-9 208-96-8 120-12-7 56-55-3 50-32-8 205-99-2 191-24-2 207-08-9 218-01-9	Acenaphthene Acenaphthylene Anthracene Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoracene Benzo(g,h,i)per Benzo(k)fluoracene Chrysene	cene : nthene rylene	ND ND ND ND ND ND ND ND	5.7 5.7 5.7 5.7 5.7 5.7 5.7 5.7 5.7	1.8 0.40 0.66 0.34 0.33 0.51 0.42 0.40	ug/l ug/l ug/l ug/l ug/l ug/l ug/l ug/l		
53-70-3 206-44-0 86-73-7 193-39-5 91-57-6 91-20-3 85-01-8	Dibenz(a,h)antl Fluoranthene Fluorene Indeno(1,2,3-co 2-Methylnaphth Naphthalene Phenanthrene	l)pyrene	ND ND ND ND ND ND ND	5.7 5.7 5.7 5.7 5.7 5.7 5.7	0.44 0.38 0.45 0.33 0.51 1.1	ug/l ug/l ug/l ug/l ug/l ug/l ug/l		
129-00-0	Pyrene C11-C22 Arom C9-C18 Alipha C19-C36 Aliph C11-C22 Arom	tics atics	ND 33.7 24.0 33.6 33.7	5.7 110 110 110 110	0.68 33 19 31 33	ug/l ug/l ug/l ug/l ug/l	JB JB J JB	
CAS No.  84-15-1 321-60-8 3386-33-2 580-13-2	o-Terphenyl 2-Fluorobiphenyl 1-Chlorooctaded 2-Bromonaphth	yl cane	Run# 1 61% 69% 52% 75%	Run# 2	40-1 40-1	its 40% 40% 40% 40%	Mé Mé	Infante sadez

ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

#### **EXECUTIVE NARRATIVE**

SDG No:

MC46948

Laboratory: Accutest,

**Accutest, Massachusetts** 

Analysis:

MADEP EPH

Number of Samples:

Location:

BMSMC, Phase 2A Release Assessment Area

Humacao, PR

SUMMARY:

Three (3) samples were analyzed for Volatiles TPHC Ranges by method MADEP EPH. Samples were validated following the METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** 

None

Major:

None

Minor:

None

**Critical findings:** 

None

**Major findings:** 

None

Minor findings:

1. Analytes detected in method blank at a concentration below the reporting limits. Analytes detected in sample batch above MDL but below the reporting limits. Laboratory qualified the results as JB, no further

qualification required.

2. Surrogate standard (1-chlorooctadecane) recovered outside control limit in sample MC46948-1. Outside control limits due to matrix interference.

Confirmed by refractionation/reanalysis. No action taken.

**COMMENTS:** 

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

August 2, 2016

Date:

# SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: MC46948-1

Sample location: BMSMC Phase 2A Release Assessment, Humacao, PR

Sampling date: 7/19/2016 Matrix: Groundwater

METHOD: 8270D

Analyte Name	Result	Units	<b>Dilution Factor</b>	Lab Flag	Validation	Reportable
Acenaphthene	5.6	ug/l	1	-	บ	Yes
Acenaphthylene	5.6	ug/i	1	-	U	Yes
Anthracene	5.6	ug/l	1	-	Ų	Yes
Atrazine	5.6	ug/l	1	-	U	Yes
Benzo(a)anthracene	0.45	ug/l	1	JB	JB	Yes
Benzo(a)pyrene	5.6	ug/l	1	-	U	Yes
Benzo(b)fluoranthene	5.6	ug/l	1	-	U	Yes
Benzo(g,h,i)perylene	5.6	ug/i	1	-	U	Yes
Benzo(k)fluoranthene	5.6	ug/l	1	-	U	Yes
Chrysene	5.6	ug/l	1	-	U	Yes
Dibenzo(a,h)anthracene	5.6	ug/l	1	-	U	Yes
Fluoranthene	5.6	ug/l	1	-	U	Yes
Fluorene	5.6	ug/l	1	-	U	Yes
Indeno(1,2,3-cd)pyrene	5.6	ug/l	1	-	U	Yes
2-Methylnaphthalene	5.6	ug/l	1	-	U	Yes
Naphthalene	5.6	ug/l	1	-	U	Yes
Phenanthrene	5.6	ug/l	1	-	U	Yes
Pyrene	5.6	ug/l	1	-	U	Yes
C11-C22 Aromatics (Unadj.)	40.5	ug/l	1	JB	JB	Yes
C9-C18 Aliphatics	19.7	ug/l	1	JB	JB	Yes
C19-C36 Aliphatics	36.5	ug/l	1	J	1	Yes
C11-C22 Aromatics (Unadj.)	38.2	ug/l	1	JB	JB	Yes

Sample ID: MC46948-2

Sample location: BMSMC Phase 2A Release Assessment, Humacao, PR

Sampling date: 7/19/2016 Matrix: Groundwater

METHOD: 8270D

Analyte Name	Result	Units (	Dilution Factor	Lab Flag	Validation	Reportable
Acenaphthene	5.7	ug/l	1	-	U	Yes
Acenaphthylene	5.7	ug/i	1	-	U	Yes
Anthracene	5.7	ug/l	1	-	U	Yes
Atrazine	5.7	ug/l	1	-	U	Yes
Benzo(a)anthracene	5.7	ug/l	1	-	U	Yes
Benzo(a)pyrene	5.7	ug/l	1	-	U	Yes
Benzo(b)fluoranthene	5.7	ug/l	1	-	U	Yes
Benzo(g,h,i)perylene	5.7	ug/l	1	-	U	Yes
Benzo(k)fluoranthene	5.7	ug/l	1	-	U	Yes
Chrysene	5.7	ug/l	1	-	U	Yes
Dibenzo(a,h)anthracene	5.7	ug/l	1	-	U	Yes
Fluoranthene	5.7	ug/l	1	-	U	Yes
Fluorene	5.7	ug/i	1	-	U	Yes
indeno(1,2,3-cd)pyrene	5.7	ug/l	1	-	U	Yes
2-Methylnaphthalene	5.7	ug/l	1	-	U	Yes
Naphthalene	5.7	ug/l	1	-	U	Yes
Phenanthrene	5.7	ug/l	1	-	U	Yes
Pyrene	5.7	ug/l	1	-	U	Yes
C11-C22 Aromatics (Unadj.)	33.7	ug/l	1	JB	JB	Yes
C9-C18 Aliphatics	24.0	ug/l	1	JB	JB	Yes
C19-C36 Aliphatics	33.6	ug/l	1	1	J	Yes
C11-C22 Aromatics (Unadj.)	33.7	ug/l	1	JB	JB	Yes

Sample ID: MC46948-3

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Sample location: BMSMC Phase 2A Release Assessment, Humacao, PR

Sampling date: 7/20/2016 Matrix: Groundwater

METHOD: 8270D

Analyte Name	Result	Units	<b>Dilution Factor</b>	Lab Flag	Validation	Reportable	
Acenaphthene	5.7	ug/l	1	-	U	Yes	
Acenaphthylene	5.7	ug/i	1	-	U	Yes	
Anthracene	5.7	ug/l	1	-	U	Yes	
Atrazine	5.7	ug/l	1	-	U	Yes	
Benzo(a)anthracene	5.7	ug/l	1	-	U	Yes	
Benzo(a)pyrene	5.7	ug/l	1	-	U	Yes	
Benzo(b)fluoranthene	5.7	ug/l	1	-	U	Yes	
Benzo(g,h,i)perylene	5.7	ug/l	1	-	U	Yes	
Benzo(k)fluoranthene	5.7	ug/l	1	-	U	Yes	
Chrysene	5.7	ug/l	1	-	U	Yes	
Dibenzo(a,h)anthracene	5.7	ug/l	1	-	U	Yes	
Fluoranthene	5.7	ug/l	1	-	U	Yes	
Fluorene	5.7	ug/i	1	-	U	Yes	
indeno(1,2,3-cd)pyrene	5.7	ug/l	1	-	U	Yes	
2-Methylnaphthalene	5.7	ug/l	1	-	U	Yes	
Naphthalene	5.7	ug/l	1	-	U	Yes	
Phenanthrene	5.7	ug/l	1	-	U	Yes	
Pyrene	5.7	ug/l	1	-	U	Yes	
C11-C22 Aromatics (Unadj.)	33.6	ug/l	1	JB	JB	Yes	
C9-C18 Aliphatics	25.2	ug/l	1	JB	JB	Yes	
C19-C36 Aliphatics	58.5	ug/l	1	1	J	Yes	
C11-C22 Aromatics (Unadj.)	33.6	ug/l	1	JB	JB	Yes	

# **DATA REVIEW WORKSHEETS**

Type of validation	Full:X Limited:	Project Number: _MC46948 Date:07/19-20/2016 Shipping date:07/20/2016 EPA Region:2
REVIEW OF EXT	RACTABLE PETROLI	EUM HYDROCARBON (EPHs) PACKAGE
validation actions. This more informed decision were assessed accord precedence METHOD HYDROCARBONS (EF (2004). Also the general Support Section. The Common section is a section of the common section.	document will assist the n and in better serving ing to the data validation FOR THE DETERIPH), Massachusetts Depral validation guidelines	ile organics were created to delineate required reviewer in using professional judgment to make the needs of the data users. The sample results on guidance documents in the following order of MINATION OF EXTRACTABLE PETROLEUM artment of Environmental Protection, Revision 1.1 promulgated by the USEPA Hazardous Wastes lation actions listed on the data review worksheets is otherwise noted.
The hardcopied (labo received has been review for SVOCs included)	ewed and the quality co	st_Laboratories data package ntrol and performance data summarized. The data
Lab. Project/SDG No.: No. of Samples: Field blank No.: Equipment blank No.: Trip blank No.: Field duplicate No.:	3	Sample matrix: _Groundwater
X Data Complet X Holding Time: N/A GC/MS Tunin N/A Internal Stand X Blanks X Surrogate Rec	teness s g ard Performance	X Laboratory Control SpikesX Field DuplicatesX CatibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Overall _Extractable_Petroleum (C9_to_C36_Aliphatics;	n_Hydrocarbons_by_GC ;_C11_to_C22_(Aromati	Comments: _by_Method_MADEP_EPH,_REV_1.1cs)
Definition of Qualifiers:		
J- Estimated resulu- U- Compound not R- Rejected data UJ- Estimated non Reviewer: A	detected	

	Criteria were not m	All criteria were metx net and/or see below
I. DATA COMPLETNE A. Data Packag		
MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
3. Other		Discrepancies:

All criteria were met	X
Criteria were not met and/or see below	

### **HOLDING TIMES**

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples	extracted and ar	nalyzed within met	hod recommende	ed bolding time
				Tolding tillo.
	<u></u>			

## Criteria

## Preservation:

Aqueous samples must be acidified to a pH of 2.0 or less at the time of collection.

Soil samples must be cooled at 4 + 2 °C immediately after collection.

### Holding times:

Samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ). If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R). If samples were not at the proper temperature (> 10°C) or improperly preserved, use professional judgment to qualify the results.

All criteria were metX  Criteria were not met and/or see below								
CALIBRAT	CALIBRATIONS VERIFICATION							
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.								
Dat	e of initial calib	ration:06/22	2/16					
Dat	es of initial cali	bration verification:_	06/22/13	****				
Inst	rument ID num	bers:GCD	E					
Mat	rix/Level:	_AQUEOUS/MEDIUI	M					
DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED				
Initia	al and initial ca	libration verification r	neet method specific r	equirements.				

## Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest.
   When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons using the FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.
  - The area for the surrogates must be subtracted from the area summation of the range in which they elute.
  - The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C9-C18 Aliphatic Hydrocarbon range area prior to calculating the CF.

# **DATA REVIEW WORKSHEETS**

#### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than ±25%, a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	_06/22/16
Dates of continuing calibration verification:	_07/25/16;_07/26/16
Dates of final calibration verification:	_07/25/16;_07/26/16
Instrument ID numbers:GCDE	
Matrix/Level:_SOIL/AQUEOUS/MEDIUM	

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
	Initial and conti	nuing calibration me	et method specific requ	uirements

A separate worksheet should be filled for each initial curve

		C	riteria were not n	All criteria were met net and/or see belowX
VA. BLAN	K ANALYSIS R	ESULTS (Se	ctions 1 & 2)	
magnitude of blanks associ problems wit evaluated to case, or if the Method Blan	contamination ( iated with the s h any blanks e determine whet e problem is an	problems. The amples, included in the case of the case	ne criteria for eva uding trip, equipn a associated with ere is an inheren currence not affect as suspected of	determine the existence and luation of blanks apply only to nent, and laboratory blanks. If the case must be carefully it variability in the data for the cting other data. A Laboratory being highly contaminated to
List the conta separately.	amination in the	blanks belov	w. High and low	ievels blanks must be treated
Laboratory bl	anks			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_CASES_DE _07/25/16	SCRIBED_IN_T OP48223-MB_ Analytes detectionits. Analyte	Aqueous/loved in methods detected in methods. Laborato	MENT	ematics_(Unadj.)_44.7_ug/l_omaticis41.9_ug/l_omaticis41.9_ug/l_omaticis21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics21.6_ug/l_omatics
Field/Trip/Equ	ipment			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_NO_EQUIPM _PACKAGE	MENT/FIELD/_A	NALYZED_A	ASSOCIATED_W	/ITH_THIS_DATA
Note:				

	All criteria were met	
Criteria were not	met and/or see below	Χ

# V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

			A	All crite	ria w	ere	met	
Criteria	were	not	met	and/or	see	belo	WC	Χ

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment. List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

Samples and QC shown here apply to the above method

Lab	Lab				
Sample ID	File ID	S1 a	\$2 <b>a</b>	S3 b	S4 a
MC46948-1	DE14962.D	57	62	36* c	70
MC46948-1	DE14944.D	65	79	35* c	85
MC46948-2	DE14945.D	61	69	52	75
MC46948-3	DE14946.D	54	64	50	69
OP48223-BS	DE14941.D	72	69	53	71
OP48223-BSD	DE14942.D	71	71	54	70
OP48223-MB	DE14943.D	75	71	59	79

Recovery
Limits
40-140%
40-140%
40-140%
40-140%

(a) Recovery from GC signal #1

- (b) Recovery from GC signal #2
- (c) Outside control limits due to matrix interference. Confirmed by refractionation/reanalysis.

Note: SURROGATE STANDARDS RECOVERIES WITHIN LABORATORY CONTROL LIMITS EXCEPT IN THE CASES DESCRIBED IN THIS DOCUMENT. NO ACTION TAKEN, PROFESSIONAL JUDGMENT.

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

# **DATA REVIEW WORKSHEETS**

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

All criteria were met	
Criteria were not met and/or see belowN/A	

# VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- Matrix duplicate Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 140% of the true value. Lower recoveries of n-nonane are permissible but must be noted in the narrative if <30%.</p>

M2/M2D Keco/	venes and Precision C	птепа			
Sample ID:	-			Matrix/Level:_	
List the %Rs, R	PD of the compounds	which do no	t meet ti	he QC criteria.	
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
	<i></i>				
As					

Note: No MS/MSD analyzed with this sample batch. Blank spike/Blank spike duplicate used to assess accuracy. BS/BSD % recoveries and RPD within laboratory control limits. No action taken.

All criteria were met	
Criteria were not met and/or see below	_N/A_

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

# 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

COMPOUND	CONCENTR SAMPLE		MSD	%RPD	ACTION
		<del></del>			

Criteria: None specified, use %RSD ≤ 50 as professional judgment.

Actions:

If the % RSD > 50, qualify the results in the spiked sample as estimate (J). If the % RSD is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.

	All criteria were metX Criteria were not met and/or see below						
VIII.	LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS						
This d	ata is generated to determine accuracy of the analytical method for various						
1.	LCS Recoveries Criteria						
	List the %R of compounds which do not meet the criteria						
LCS ID	COMPOUND % R QC LIMIT ACTION						
LCS_RECO	OVERY_WITHIN_LABORATORY_CONTROL_LIMTS						
*  *  Action:	<ul> <li>* Refer to QAPP for specific criteria.</li> <li>* The spike recovery must be between 40% and 140%. Lower recoveries of n-nonane are permissible. If the recovery of n-nonane is &lt;30%, note the nonconformance in the executive narrative. RPD between LCS/LCSD must be &lt; 25%.</li> <li>Actions: Actions: Actions on LCS recovery should be based on both the number of compounds that are outside the %R and RPD criteria and the magnitude of the excedance of</li> </ul>						
If the %R of the associated if the %R of the affected if more than h	ne analyte is > UL, qualify all positive results (j) for the affected analyte in a samples and accept nondetects.  The analyte is < LL, qualify all positive results (j) and reject (R) nondetects analyte in the associated samples.  The all the compounds in the LCS are not within the required recovery criteria, itive results as (J) and reject nondetects (R) for all target analyte(s) in the						
2. Freque	ency Criteria:						
per matrix)? Y If no, the data the effect and	nalyzed at the required frequency and for each matrix (1 per 20 samples es or No.  may be affected. Use professional judgment to determine the severity of qualify data accordingly. Discuss any actions below and list the samples uss the actions below:						

	All criteria were metX Criteria were not met and/or see below						
IX. FIELD/LA	X. FIELD/LABORATORY DUPLICATE PRECISION						
Sample IDs:	MC4694	8-1/MC46948-2	Matrix:	Gro	undwater		
Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.							
COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION		
Field duplicate analyzed with this data package. RPD within laboratory and validation guidance document control limits (± 50 %) for analytes detected at a concentration > SQL.							

### Criteria:

The project QAPP should be reviewed for project-specific information. RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples if results are  $\geq$  SQL. If both samples and duplicate are  $\leq$ 5 SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5x$  the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is < 5x the SQL, use professional judgment to determine if qualification is appropriate.

All criteria were metX	
Criteria were not met and/or see below	

### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TiCs).

- 1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target EPH
     Analyte each time a new GC column is installed, and must be verified
     and/or adjusted on a daily basis.
  - o The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.
  - o All surrogates must be adequately resolved from the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.
- 1a. Aliphatic hydrocarbons range:
  - o Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C9 and 0.01 minutes before the Rt for n-C19.
  - o Determine the total area count for all peaks eluting 0.01 minutes before the Rt for n-C19 and 0.1 minutes after the Rt for n-C36.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

- 1b. Aromatic hydrocarbons range:
  - Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.
  - Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract these values from the collective area count value.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

Comments: Not applicable.

		0 11		vere metX
		Criteria wer	e not met and/or s	see below
2.	If target analytes ar laboratory resubmit the	nd/or TICs were not com ne corrected data.	ectly identified, r	request that the
3.	evaluated for potentia % recovery of the fra basis by quantifying and aromatic fraction naphthalene or 2-methe total concentration LCSD, fractionation	nination - Each sample all breakthrough on a sample actionation surrogate (2-br naphthalene and 2-methyns of the LCS and LCSD ethylnaphthalene in the action for naphthalene or 2 on must be repeated on	le specific basis be comonaphthalene) Inaphthalene in be if either the co- aliphatic fraction I-methylnaphthal all archived batc	oy evaluating the and on a batch oth the aliphatic oncentration of exceeds 5% of ene in the LCS h extracts.
	NOTE:	The total concentrate methylnaphthalene in the summation of the coaliphatic fraction and the aromatic fraction.	he LCS/LCSD pa oncentration de	tected in the
	Comments:Concer _concentration_for_n	ntration_in_the_aliphatic_fr aphthalene_and_2-methyl	action_<_5%_of_ naphthalene	the_total
4.	containing 14 alkane each constituent. The fractionation efficience optimum hexane volunot allowing signification tailouing in the fractional contained in the fractional contained in the significant contained in the sig	k Standard – A fractional stand 17 PAHs at a nominal Fractionation Check Solution of each new lot of silical time required to efficiently count aromatic hydrocarbonationation check solution, etween 40 and 140%. A 36	inal concentration tion must be used gel/cartridges, a elute aliphatic hyd breakthrough. Fo excluding n-nona	ion is prepared of 200 ng/µl of to evaluate the nd establish the lrocarbons while or each analyte ne, the Percent
	Is a fractionation chec	ck standard analyzed?		Yes? or No?

All criteria were met	Χ
Criteria were not met and/or see below	

# XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative.

The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

Is aliphatic mass discrimination observed in the sample?

Yes? or No?

Is aromatic mass discrimination observed in the sample?

Yes? or No?

1. In the space below, please show a minimum of one sample calculation:

MC46948-1

EPH (C11 – C22, Aromatics)

RF = 124800

[] = (2248458)/(124800)

[] = 18.02 ppb Ok

MC46948-1

EPH (C19 – C36, Aliphatics)

RF = 77820

[] = (1265664)/(77820)

[] = 16.26 ppb Ok

# **DATA REVIEW WORKSHEETS**

- 2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
- 3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION		
	2			
		<del>-</del>		
		V 3 - 67 7 1 6 17 1		
	1	A 5.00 (A. A. )		
	2 22			

If dilution was not performed, affected samples/compounds:	results	(J) fo	or the	affected	compounds.	List the
				····		